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MONTHLY PROGRESS REPORTS

Period Covered

November 1 - 30, 1973

2022160798

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MONTHLY PROGRESS REPORTS

Period Covered

November 1 - 30, 1973

Date Issued: December 15, 1973

2022160799

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2022160800

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CHARGE NUMBER: Various

PROGRAM TITLE: Analytical Investigations

WRITTEN BY: M. C. Bourlas, D. C. Watson, and E. T. Oakley

DATE OF REPORT: December 13, 1973

I. PYROLYSIS OF TOBACCO

The FTS-14 infrared spectrometer was employed to monitor gases generated by heating tobacco from ambient to 1000°C. The IR spectra were recorded every 10°C. The spectra revealed qualitative as well as quantitative changes in the gaseous components. The CO and CO_2 concentration profiles from this work correlated well with data obtained previously. The concentration profiles were recorded also for methane, ethylene, methanol, ammonia, and isoprene.

The latter was studied also using hexane extracted and non-extracted tobaccos. The preliminary data indicated that the isoprene concentration at 350°C decreased sharply when hexane extracted tobacco was used.

II. TOTAL AMINO ACIDS IN TOBACCO

A literature method utilizing the reaction of dinitrofluorobenzene with amino acids is being automated on the Robot Chemist. Aspartic acid is being used as a standard, proline having a 30% greater response and asparagine a 40% lower response when all are 100 ppm alone in a standard solution. Development of this method would give us the capacity to estimate total amino acids by a rapid colorimetric procedure.

III. ALDEHYDES AND NITRILES

Ratios of TPM cyanide to gas phase cyanide has been determined for the following tobaccos:

M #16	0.52
All burley	0.67
All bright	0.58
All Turkish	0.34
Kentucky Reference	0.50

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IV. SIDESTREAM NICOTINE AND PHENOLS

Smoking and collection procedures have been optimized and data collected on M#16. The "chimney" designed by R. Hale is being used and SS collected on a Cambridge pad followed by an acid trap. A second base trap has been eliminated because it was found that only 2% of the total SS phenol went through the first trap. An aliquot of the collection trap can also be used for SS ammonia analysis. Results will be reported as SS/MS ratios.

V. SMOKE COMPONENT SEPARATION

Several capillary columns have been prepared for use in smoke component separation. Column efficiency is good for the gas phase and volatile portions of smoke. Experiments with sample introduction systems indicates the Tenax trap (Ref. A. Zlatkis) to be an efficient means of concentrating smoke components.

/np

Manuel C. Bowles

2022160802

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PROJECT NUMBER: 0105

PROJECT TITLE: PHYSICAL CHEMISTRY OF SMOKE AND FILTRATION

PERIOD COVERED: November 1 - 30, 1973

DATE OF REPORT: December 14, 1973

PROJECT LEADER: H. A. Hartung

Sorption in Filters

This month has been devoted largely to organization and reduction of data, literature studies and planning. Experiments were completed on the adsorption of alkanes and alkenes on cellulose acetate over the temperature range of 31 to 85°C. There are indications of unexpected physical transitions or changes as the CA is heated up. This is a complication that interferes with plans to measure heats of adsorption and we have done some detailed work in an attempt to elucidate both factors.

Baseline Studies

The study of Marlboro characteristics as a function of moisture is proceeding smoothly. The cigarettes were equilibrated for over a month in at eleven different target moisture contents. Oven volatiles (OV) have come in very close to the target values. Static burn times were found to be a linear function of the OV.

Isolation and Study of Flavor Constituents

Work during this month was devoted mostly to completing the large scale tobacco sparging unit and conducting experiments with the "mini-sparging" apparatus coupled with the Fourier transform infra-red instrument. The latter experiments were aimed at determining the rates of evolution of various substances from tobacco as it is heated in a stream of inert gas. The experiments were quite successful and much preliminary data was obtained (1).

/gd

References:

(1) Osborne, Notebook 6143; pp. 41-42.

H. A. Hartung

H. A. Hartung

2022160803

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CHARGE NUMBER: 0108

PROGRAM TITLE: MECHANISM FOR SMOKE FORMATION

PERIOD COVERED: NOVEMBER 1-30, 1973

PROJECT LEADER: W. R. JOHNSON

DATE OF REPORT: DECEMBER 6, 1973

I. MENTHOL RELEASE POLYMERS[1-5]

Poly(vinyl *l*-menthyl carbonate) was prepared by the reaction of potassium menthoxide with vinyl chloroformate and subsequent polymerization of the monomer with benzoyl peroxide. The polymer was also prepared by the reaction of the menthoxide with poly(vinyl chloroformate). Both methods gave products which possessed about 1% chlorine. Otherwise, physical data indicate that the desired polymer was obtained. Calculations show that the degrees of menthol substitution obtained by the two routes exceed 0.9.

A large sample (4g) of poly(α-styryl *l*-menthyl carbonate) was prepared by polymerization of the monomer using benzoyl peroxide. The polymerization was carried out in the presence of the previously reported triphenylmethane impurity which was successfully removed from the finished polymer.

II. DIFFUSION STREAM STUDIES[6-8]

Several components of the diffusion stream were reported last month to be present in concentrations which are independent of paper porosity over the 11.8 to 36.4 sec range. Carbon dioxide has been found to give increasing concentrations with increasing paper porosity. Carbon monoxide concentrations, on the other hand, decrease with increasing porosity. This decrease is thought not to involve different diffusional behavior but is thought to result from alterations in the chemical reactions at the coal. Such alterations are most likely consequences of the oxygen supply at the coal which varies inversely with air flow through the cigarette paper during the puff.

Ratios of the diffusion stream concentrations of various gas phase components obtained from normal puffing cycles to the concentrations obtained when cigarettes are consumed statically show components to respond to changes in smoking conditions

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quite differently. The ratios were 0.9, 2, 15 and 32 for nitrous oxide, carbon dioxide, carbon monoxide and acetonitrile respectively. These differences in behavior corroborate the findings of a recent study of mainstream and sidestream smoke which show that the concentrations of some smoke components (*e.g.*, methyl chloride) in the two streams are essentially functions of tobacco consumption, while the concentrations of others (*e.g.*, hydrogen cyanide, ammonia) do not follow consumption data at all, but appear to be dependent upon different formation conditions which exist in the two streams.[9]

III. PUBLICATION

W. R. Johnson, R. W. Hale, J. W. Nedlock, H. J. Grubbs, and D. H. Powell, "The Distribution of Products Between Mainstream and Sidestream Smoke," Tobacco Science, 17, 141-144 (1973).

IV. REFERENCES

1. H. Grubbs 6420/22-34
2. T. Van Auken 6306/73-86
3. L. Morgan 6366/48,52
4. M. Dodson 6431/40,43,46,48,51
5. G. Vilcins 6431/31
6. D. Powell 6097/3-7
7. R. Hale 6214/74-89
8. L. Brown 6331/21-24
9. W. R. Johnson, R. W. Hale, J. W. Nedlock, H. J. Grubbs, and D. H. Powell, "The Distribution of Products Between Mainstream and Sidestream Smoke," Tobacco Science, 17, 141-144 (1973).

W. R. Johnson

/slb

2022160805

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CHARGE NUMBER: 1101

PROGRAM TITLE: ENTOMOLOGICAL RESEARCH

PERIOD COVERED: NOVEMBER 1 - 30, 1973

PROJECT LEADER: M. A. MANZELLI

DATE OR REPORT: DECEMBER 4, 1973

I. CIGARETTE BEETLE GROWTH ON VARIOUS CULTURE MEDIA

A series of beetle cultures incorporating various P.M. tobaccos as media was initiated in August for the purpose of demonstrating the effects of the latter on cigarette beetle growth and development.[1] Final results were as follows:

CULTURE MEDIUM	DAYS TO INITIAL ADULT EMERGENCE	DAYS TO FINAL ADULT EMERGENCE	AVER. NO. OF BEETLES PER CULTURE	AVER. WEIGHT PER BEETLE IN MGS.
Flour-yeast control	31	60	2582	2.17
Bright	60	120	94	1.14
Burley	68	120	17	-- (a)
Turkish	60	120	48	1.00
RCB	70	99	18	0.92
RKS	60	120	153	1.41
ET	69	120	88	1.07
Filler	60	120	127	1.10

(a) Insufficient number of beetles available at any one time to permit significant weighing.

Burley tobacco has never appeared to be too attractive to the cigarette beetle, and the above data reinforces this belief. RCB is equally unattractive in this respect. There is no questioning the superiority of the flour-yeast medium over the tobacco media for the production of vigorous colonies of the beetle. RKS, Bright, filler and ET tobaccos rank in that order in decreasing attractiveness to the cigarette beetle.

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II. EFFECT OF SOME ACIDS ON THE GROWTH OF THE CIGARETTE BEETLE

The growth inhibition effect of adding 1% levels of the following acids to the flour medium employed to culture the cigarette beetle is shown below:[2]

MEDIUM AND ACID	NO. OF ADULT BEETLES DEVELOPED/JAR (a)
Flour and yeast control	1913
Flour	959
Flour and 1% sorbic acid	24
Flour and 1% citric acid	965
Flour and 1% malic acid	887
Flour and 1% propionic acid	49

(a) Each jar inoculated with 100 adult beetles

The decline in beetle population brought about by the action of sorbic and propionic acids was not unexpected. Both of these acids possess fungicidal activity, and apparently they eliminated the yeast symbionts found within the midgut of the cigarette beetles and which function as producers of essential B complex vitamins.

III. REFERENCES

1. R. Lehman 6186/47-53, 74-75
2. R. Lehman 6186/45-46, 54-56

/slb

M. A. Mangelli

2022160807

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CHARGE NUMBER: 1300

PROGRAM TITLE: Sheet Materials Development

PROJECT LEADER: G. Gellatly

PERIOD COVERED: November 1-30, 1973

DATE OF REPORT: December 11, 1973

I. RCB

The results of sheet weight and piece size trials indicate that there may be come improvement in coal strength of a normal MF blend by increasing the sheet weight from 12 gms/ft^2 to 14 gms/ft^2 and decreasing the piece size from 4 inch squares to 3 x 4 inch pieces. The results, however, are very variable and many more trials would have to be run before a definite recommendation was made. Variation of sheet weight and piece size had no apparent effect on cylinder volume.

B.L. Pilot Plant trials to make RCB from Turkish scrap indicate that some reduction of tensile strength is being caused by the high sand content of the raw material. Trials will be run on desanded scrap to substantiate this theory. A Turkish RCB of 0.5 Kg/in tensile strength has been achieved so far which is considered satisfactory.

II. SCHWEITZER TOBACCO SHEET

Production runs of SRT Nos. 7, 8 and 9-73 were found to be within specification for solubles, potassium sorbate and TEG. A production run of SBS No. 10-73 was made at Spotswood and is being assessed.

The light color of SBS produced at LeMans may be explained by the low solubles level (1%) of the web to the Yankee Dryer. Spotswood, New Jersey's web has a solubles level of 10% and produces much darker SBS sheet. This phenomenon will be investigated when the RL Pilot Plant is operational.

No significant difference was found between cigarettes containing cut and threshed SRT at 17.5% level in an MF blend substituting RCB. Cigarettes were compared on the basis of coal strength, sieve analysis and other cigarette parameters. Threshing the RL was recommended to Manufacturing rather than cutting because of lower capital and maintenance cost of equipment.

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III. RGL

Trials were initiated to convert the starch content of bright green leaf to sugar by phosphonic acid treatment prior to DAP treatment.

IV. NITRATE REDUCTION OF BURLEY STEMS

Work is proceeding to quantify nitrate removal from burley stem liquor by crystallization.

V. E&R PILOT PLANT

Installation is anticipated before the end of the first quarter of 1974. The delivery date for the continuous pulper and one batch pulper has been extended to March 1 by the manufacturers. However, it is feasible that we can run the other batch pulper in a continuous mode to prove out equipment while we await this delayed equipment.

G. Gellatly

/jl

2022160809

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CHARGE NUMBER: 1503

PROGRAM TITLE: Modified Smoking Materials

PROJECT LEADER: J. D. Hind

PERIOD COVERED: November 1-30, 1973

DATE OF REPORT: December 11, 1973

I. FILM WRAPPERS[1]

The Sandvik belt will not be available to us for six to eight weeks, pending installation of RL process equipment in "C" Pilot Plant. Before this shutdown, we prepared several new film wrapper formulations using guar gum with additions of alumina pigment and potassium citrate respectively.

We are testing these films and making others in the laboratory to define the effects which can be obtained by making wide variations in amount and kinds of pigment loading.

Rewinding equipment to permit uniform distribution of moisture in the films is being constructed in the machine shop. The New Product Division (Project 4009) is preparing various model cigarettes from guar gum wrapper and will be working on mechanically perforated films.

In guar wrapper, Players (all flue cured) blend delivers less TPM and moisture from Marlboro 85 models than MF blend.

II. FILLER PROCESSES

Redried ammonium carbonate impregnated filler was successfully expanded[2] in the 3" tower at a temperature of less than 250°F. Cylinder volume attained was 71 cc/10 g at 11% O.V.

We have observed[3] that uncased burley tobacco filler can be expanded to high cylinder volumes in the 3" tower operating at 625°F, 100% steam, 100 ft/sec. Input tobacco was adjusted to 20 and 30% O.V. The tobacco had been specially cut for experiments with ammonia-hydrogen peroxide, but it was apparent that these additives were not necessary to obtain the desired expansion. A systematic series of tests is underway to determine whether these preliminary findings can be reproduced in large-scale runs.

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Although members of the in-house smoking panel apparently did not detect off-taste in an initial trial of aluminum-sulphate cased filler, some objections appeared in a descriptive panel trial. Our Flavor Group has recommended an additive to be used with this filler.[4]

1-3 butanediol has been tried on Marlboro type cigarettes at approximately a 3% level withholding other casings from the trial. This humectant has been used extensively in Germany where it is an accepted cigarette ingredient. The Flavor Group has been requested to evaluate the cigarettes.[4]

TFP, with 14% carbon additive was treated in the 3" tower, in an attempt to reactivate the carbon. Appropriate blends are being made into cigarettes to evaluate this treatment. The carbon addition is part of a systematic effort to find extenders for reconstituted material which may result in an overall improvement of our blends.[4]

III. DESIGNED FILLER

Cigarettes were handmade from a composition containing 59% reduced viscosity guar gum (General Mills). The smoke was somewhat "sharp" and "papery", but there was no characteristic taste and no after-taste sensations. Guar gum can be considered from this point of view as an attractive substrate.[5]

IV. REFERENCES

[1]E. J. Deszyck, Notebook 6293, pp. 33 - 48.

[2]J. W. Leik, Notebook 6233, pp. 76 - 87.

[3]G. F. Kite, Notebook 6430, pp. 6 - 8.

[4]A. T. Lendvay, Notebook 6367, pp. 40 - 50.

[5]J. D. Hind, Notebook 4803, pp. 96.

J. D. Hind

/j1

2022160811

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CHARGE NUMBER: 1504

PROGRAM TITLE: Designed Filler

PROJECT LEADER: G. D. Keritsis

PERIOD COVERED: November 1-30, 1973

DATE OF REPORT: December 11, 1973

I. LOW TAR FILLER (LTF)

Two new cigarette models with 50% LTF in the blend were made and smoked.[1] The Descriptive Panel found both cigarettes to lack basic sweetness and be harsh to the throat. Generally, both cigarettes profiled similarly to Marlboro.[2]

The cigarettes were remade with the addition of a basic sweetener and the elimination of the additive which was believed to be responsible for the throat harshness.[3,4] These cigarettes are now being smoked.

Optimization of the 1972-LTF formulation has begun. The levels of two of the formula components were adjusted.[4] It was felt that the levels of these components was high and adversely affected the subjective smoke character.

II. NATURAL BASE FILLER (NBF)

A homogeneous sheet of 50% NBF - 50% MF was made.[5] The acceptability of the cigarettes that contained this filler was judged by the Lab Panel to be between that of Carlton and Kent cigarettes.[6]

III. CARBONIZED FILLER

Two approaches for the elimination of "fiery" particles of the carbonized filler cigarettes appear to be promising. These are now being scaled-up for a larger evaluation.[7]

IV. COMPETITIVE PRODUCTS

Planet cigarettes (U.K.) which contain 47% of Courtaulds' nontobacco filler in their blend were received and are being analyzed.

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V. DESIGNED FILLER PROCESSING

The CP-mixing equipment which is needed to foam the DF-sheet is being dismantled at Milprint. As soon as they are received, our studies will begin.

A wet-spinning technique for the production of a nontobacco smoking material was tried and appeared to be promising.[8] A small unit is now being built to further investigate this approach.

VI. REFERENCES

[1]Notebook 6123, pp. 96 - 98.

[2]Notebook 6229, pp. 24 - 26.

Notebook 6123, p. 98.

[3]Notebook 6395, p. 47.

[4]Notebook 6315, pp. 69 - 72.

[5]Notebook 6315, pp. 67 - 68.

Notebook 6439, pp. 1 - 2.

Notebook 6240, pp. 94 - 96, 98 - 99.

[6]Lab Panel Results of November 12, 1973.

[7]Notebook 6428, pp. 39 - 49.

[8]Notebook 6123, pp. 99 - 100.

G. D. Keritsis

G. D. Keritsis

/jl

2022160813

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CHARGE NUMBER: 1600

PROGRAM TITLE: Smoker Psychology

PROJECT LEADER: W. L. Dunn, Jr.

PERIOD COVERED: November 1 - 30, 1973

DATE OF REPORT: December 12, 1973

Project Title: Alpha Brain Waves and Smoking
Written by: W. L. Dunn

We are continuing to collect data in Phase I.

Project Title: Controlling Smoke Inhalation Post-Puff
Written by: W. L. Dunn

Having decided that the three essential measures at this stage of the study are nasal air inhalation volume, oral air inhalation volume and puff volume, we are actively investigating the various ways of obtaining these measures under conditions of minimal obtrusiveness. Tentatively we have settled upon a space-helmet approach using a mass flow meter (thermistor,. sometimes called hot-wire anemometer) to measure nasal and oral air volumes. We anticipate differences in these measures and in the temporal patterning of these flows among individuals and changes for a given individual as a function of changes in smoke composition and/or concentration. Since nasal and oral air flows are rarely measured discretely in respiratory investigations, considerable innovation in and customizing of instrumentation is likely to be needed.

Project Title: Frustration and Smoking (Echo)
Written by: Frank Ryan

We are now designing a study, using the delayed auditory feedback tape recorder, to investigate whether smokers will be affected less after smoking than after not smoking when reading under delayed feedback or immediate feedback conditions.

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Project Title: Puffing Behavior
Written by: Frank Ryan

Data gathering is continuing on the present study, Puff Five. This study is investigating the effects of five different cigarette lengths on puff volume, flow rates, and puff duration.

Project Title: MX Studies
Written by: Frank Ryan

The RP^3 panel found no significant differences between packs of cigarettes containing either 7.6 mg or 12.8 mg FTC tar and packs containing both 7.6 mg and 12.8 mg cigarettes in a 50-50 mix (two two-pack handouts were tested).

Project Title: Smoking, Arousal, and Mood Change
Written by: T. R. Schori

We have been running college students as pilot subjects on this study. And we will now commence running regular subjects. The data acquisition phase should continue for approximately 3 months.

Project Title: Tar, Nicotine, and Smoking Behavior (TNT-4)
Written by: T. R. Schori

The technical report for this study should soon be ready for distribution.

Project Title: Miscellaneous
Written by: T. R. Schori

We are currently awaiting cigarettes for two mailout studies--DL-1 and MN-3. And we are in the process of requesting cigarettes for a third mailout study--DL-2. Our paper "Rorschach Factors and Somatotype" was published in the Journal of Clinical Psychology.

/jlh

W. L. Dunn

2022160815

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Project Number: 1706

Project Title: Tobacco Physics

Period Covered: November 1 - November 30, 1973

Written by: A. C. Lilly

Date of Report: December 7, 1973

Conversion of Rod to Smoke[1,2,3,4]

Work has continued on the interpretation of the experimental data obtained on the output of Marlboro Monitor #15 cigarettes over a wide range of parameters. In particular smooth functions have been to the TPM-flow rate data for the unfiltered rod and for the rod with several common types of filters. These functions are as follows:

Cigarette	TPM/35cc Puff (F is Flow Rate)
Marlboro M15 without filter	$1.242 + 5.191\ e^{-.000197F}$
Marlboro M15 with Marlboro CA filter	$2.683F^{.03307}e^{-.0001163F}$
Marlboro M15 with Ecusta Paper Filter	$1.098\ F^{.1417}e^{-.0001836F}$
Marlboro M15 with Dilution CA filter	$.5911\ F^{.2188}e^{-.0001457F}$
Marlboro M15 with Filtrona filter	$.4828\ F^{.2846}e^{-.0001634F}$

The puff volume-TPM relationship is that the TPM/puff is proportional to $(Vol)^{1.24}$ for all flow rates. Similar types of functions are being developed for tar, nicotine, and water so that they can be used for design purposes.

Work has continued on the deposition and calibration of differential thin-film thermocouples to be used in the high speed thin foil scanning calorimeter being constructed.

Catalyst Mechanism[4]

The computer program to calculate the energy levels of Cu^{2+} ions in arbitrary symmetry sites has been updated. Calculations of the crystal field levels indicates that for the octahedral and tetrahedral sites equal values of the crystal field parameters results in the levels coinciding with the observed optical bands. The pure octahedral and tetrahedral configurations give rise to fewer levels than actually observed. Distortions of geometry will be necessary to account for the observed number of transitions in the optical spectra.

/gd

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References:

(1) A. C. Lilly, Book 6292, pp. 20-35.
(2) S. L. Thurston, Book 6233, pp. 41-100.
(3) C. O. Tiller, Book 6424, p. 2-10.
(4) A. J. Kassman, Book 6274, pp. 58-67.

C. Lilly

A. C. Lilly

2022160817

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CHARGE NUMBER: 1708

PROJECT TITLE: CIGARETTE MATERIALS SCIENCE

PROJECT LEADER: N. B. Rainer

PERIOD COVERED: November 1 - 30, 1973

DATE OF REPORT: December 14, 1973

In the continued study of the chemistry of oxidized cellulosic materials, it has been discovered that oxidation of α-cellulose with ozone produces traces of oxalic acid. This result is not obtained when using NO_2 or hot air as the oxidizing agent. The amount of oxalic acid produced ranges from 0.8% with 10 hours of O_3 treatment, to 1.6% after 40 hours treatment. The ratio of insoluble to soluble (oxalic) carboxyl groups was observed to increase with severity of O_3 treatment, and can be modified by chemical and/or physical pretreatments of the cellulose. This information is expected to provide further insight into the mechanism of the reaction.[1]

Flax paper was found to react with ozone about 2.5 times faster than the reaction of α-cellulose with ozone.[2] It has also been found that potassium citrate, present in burley stem material and cigarette wrap paper, is degraded by ozone to oxalate, which in turn undergoes further degradation.[3]

A sample of cobalt/alumina catalyst for CO oxidation was evaluated by the Mine Safety Appliance Co. (MSA), and was found to be of interest for use in gas mask cannisters.[4] Further samples of the material are being prepared for shipment to MSA.

Representatives of the Hercules Chemical Company visited to learn more about our technology involving the use of microporous polystyrene to opacify paper. The Nicolet Division is also interested in this subject,[5] and samples are being prepared for their evaluation.

/gd

N B Rainer

N. B. Rainer

1. Notebook 6393, pp. 19-20; 24, 26, 29, 39.
2. Notebook 6393, p. 40.
3. Notebook 6119, pp. 83-84.
4. Letter, MSA to H. K. McMath, dated 11/27/73.
5. Letter, Nicolet to N. B. Rainer, dated 12/5/73.

2022160818

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CHARGE NUMBER: 1801

PROGRAM TITLE: Tobacco Processing

PROJECT LEADER: F. V. Utsch

PERIOD COVERED: November 1-30, 1973

DATE OF REPORT: December 11, 1973

I. IMPREGNATION STUDIES

A. Water Impregnation

Preliminary data shows that good filling power (C.C.V. at 12% O.V. = 55-60 cc/10 g) can be obtained by Jetstream expansion of MF bright impregnated to a 1:1 ratio with a hot water spray at atmospheric pressure. The impregnated material had good handling characteristics with little clumping. MF bright impregnated to a 1:1 ratio with a hot spray of 100 ppm L-77 surfactant in water gave similar expansion results. The material was perhaps slightly easier to dry indicating that the surfactant increases the rate of water removal as well as the rate of penetration. Tests are continuing.

A series of MF burley water expansion trials were conducted at feed O.V.'s of 12, 20, 30 and 40% using the 3" tower. These runs are a continuation of previous tests initiated by the Modified Smoking Materials group where encouraging results were achieved. Further tests are planned.

B. Expansion With CO_2

Impregnated filler bulking studies indicate that bulking under "Manufacturing" operational modes is feasible with hold times of 2-3 hours not unreasonable if the bulker is insulated. Cigarette compacimetric tests indicate that dry ice expanded tobacco (D.I.E.T.) is approximately equivalent to ET in filling power characteristics at the 5, 10 and 15% addition levels in MF. Preliminary Flavor Development Group subjective tests indicate that D.I.E.T. is at least equally acceptable compared to ET at the same addition levels. Further compacimetric and subjective testing is planned.

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Chemical studies show that the CO_2 process alters the filler composition to a lesser degree than the ET process with significantly less of a reduction in alkaloid and reducing sugars. Tests to evaluate the effect on C.V. of steam concentration in the expansion tower, rapid reordering, feed O.V., tower product O.V., and vacuum degassing of filler prior to impregnation have been conducted. Initial tests to study burley expansion with this process were also run.

II. TOBACCO DRYING PROCESS STUDIES

A. Jetstream and Closed-Loop Pilot Adt Dryers

The high thruput filler drying run to evaluate equipment scale-up factors was successfully conducted at 4000#/hr thruput. The run duration was limited to about 30 minutes by the amount of material available for the run. Product C.V.'s and sieve fractions are being determined. Recommendations for any further testing will be based on these results.

Three test runs of 1000# each were processed through the pilot Adt dryer and gave an average C.C.V. of 41.2 cc/10 g. After-cut flavors, ES and ET have been added and reduced-weight equal-firmness cigarettes prepared for compacimetric and RPPP testing. The C.V. profile for each process step is also being evaluated.

B. Tobacco Dryer Fundamental Studies

A report is being issued which describes the computer program written to predict the actual gas flow through the dryer. The report includes parametric studies performed using the program and thereby establishing the accuracy of the calculation procedure. The measurement of the dryer exit gas temperature is necessary in the application of this program and has not yet been achieved.

A computer program has been written to predict the tobacco temperature and oven volatiles as a function of dryer length. Work is being done to achieve correlations with data gathered from the pilot Adt. Progress has been made in establishing drying rate constants and tobacco/air transfer coefficients for conditions in which only direct drying (i.e., no steam in the steam coils) occurs. The program is currently being modified to include both direct (air) and indirect (steam coils) heating.

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Source: https://www.industrydocuments.ucsf.edu/docs/slxm0000

III. REFERENCES

[1]Notebook 6301, pp. 88 - 100.

[2]Notebook 6378, pp. 70 - 100.

[3]Notebook 6447, pp. 1 - 25.

[4]Notebook 6397, pp. 26 - 34.

F. V. Utsch

F. V. Utsch

/jl

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Source: https://www.industrydocuments.ucsf.edu/docs/slxm0000

CHARGE NUMBER: 1802

PROGRAM TITLE: Stem Processing

PROJECT LEADER: L. R. Turano

WRITTEN BY: J. M. Whitman

PERIOD COVERED: November 3 - December 6, 1973

DATE OF REPORT: December 11, 1973

I. EXPANDED STEM

A. Stem Buildup - 8" Tower

The funnel feed section has been installed. The following tests have been run:

ES - Two runs have been completed. One employed a deflector in the lower elbow; the second had the deflector and the thermocouple prior to the elbow removed. In both cases, no buildup occurred in the feed section. The buildup did occur in the elbow in both runs, although removing the deflector shifted the buildup to a higher point.

ET - After two hours of running, the feed bridged in the funnel. Another funnel with a 1" larger exit (3 7/8" vs. 2 7/8") will be fabricated and the test repeated.

It should be noted that based on observations of the FTR, Louisville and Westab towers, the buildup occurring in the elbow of the 8" tower is unique. The material found in the elbows of the production towers appears to be primarily large segments which have broken loose from the buildup in the feed section. The 8" tower has approximately 1/10 the cross-sectional area of these towers (24" diameters) and has only approximately two feet from the inlet feed point to the first elbow; therefore, the material is still high in moisture and has a tendency to stick to the lower elbow upon impact.

B. Expanded Burley Stem

A program for determining optimum conditions for expanding burley stems is being carried out using the pilot plant RKS line and the 8" tower. The parameters being evaluated are: rolled

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Source: https://www.industrydocuments.ucsf.edu/docs/slxm0000

thickness, % moisture prior to the rollers, and cuts/inch. The products are being evaluated on the basis of degree of expansion and sieve analysis.

The tests completed to date have shown that a severe dust problem exists in the products expanded at the lowest rolled thickness (25 mm) and moisture levels (25-30%).

II. NH_3/CO_2 ET STUDIES

A. Expanded Tobacco - Pilot Plant

The air leg separator was used to determine the degree of ball formation using bottom addition of both NH_3 and CO_2 and forward rotation only of the agitator. The % balls found was 0.082--the worst value obtained under any conditions to date.

A report is being written recommending use of the automatic reversing cycle on the agitator and the bottom addition of ammonia only as the best methods of reducing ball formation in Manufacturing (Westab).

B. Working Model Plexiglass Impregnator

A program using lower sieve fractions of bright tobacco to facilitate loading and unloading has been set up.

Water leaks around the discharge ports have prevented carrying out successful experiments. Modifications have been made to the caps for these ports and the tests will be repeated.

III. REFERENCES

Notebook 6365.

J. M. Whitman

/jl

2022160823

Source: https://www.industrydocuments.ucsf.edu/docs/slxm0000

CHARGE NUMBER: 1901

PROGRAM TITLE: BIOCHEMICAL MODIFICATION OF TOBACCO

PERIOD COVERED: NOVEMBER 1-30, 1973

PROJECT LEADER: D. M. TENG

DATE OF REPORT: DECEMBER 7, 1973

I. WHOLE SMOKE ASSAY[1,2,3]

Work has been continued to adapt the E. coli bioassay for testing the effect of whole cigarette smoke. AD and 1R1 cigarettes as well as cigarettes made from green tobacco (freeze dried and equilibrated), flue-cured and air-cured tobacco were tested. There are no mechanical problems in adapting the E. coli assay for testing whole smoke. The only problem witnessed is that two cigarettes of the same type (same blend, RTD, weight and moisture content) smoked with the same smoking machine for the same number of puffs do not yield the same size butts. The difference is so obvious that some kind of correction is required so that different tobaccos can be compared.

Work has been continued to generate data so we can compare cigarettes made from different tobaccos by:

A. TPM

B. Same number of puffs

C. On same weight basis

D. Smoke to same butt length

This work will be reported when enough data are gathered.

II. GREEN SLURRY FERMENTATION[2]

A microfermenter was received during the last part of this month. It is now operational and will be used for determining the conditions for green tobacco slurry fermentation.

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Source: https://www.industrydocuments.ucsf.edu/docs/slxm0000

III. REFERENCES

1. D. Teng 6237
2. B. Semp 6343
3. B. Wright 6337

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CHARGE NUMBER: 2100
PROJECT TITLE: Smoke Filtration
PERIOD COVERED: November 1 - November 30, 1973
PROJECT LEADER: P. N. Gauvin
WRITTEN BY: P. N. Gauvin
DATE OF REPORT: December 12, 1973

I. New Cigarette Development

A. Exposed Plastic 100's

RP[3] testing of an exposed plastic 100 mm cigarette with white ring-tipping indicated that the appearance of the cigarette was rated significantly high on a 1-5 point scale ranging from "well below average appearance" to "well above average appearance". After smoking, the cigarette appearance was rated on a 1-9 point scale ranging from "dislike extremely" to "like extremely" and judged to be between "like slightly" and "like moderately".

The feel of the plastic filter in the mouth was acceptable.[1]

Regular white-tipped Marlboro 100's are in RP[3] testing. Initial results indicate the regular Marlboro 100's to be rated equal to the exposed plastic 100 in appearance.

SEF screening of various printed ring-tippings was completed. Instead of testing the preferred ring-tipped exposed plastic 100 mm cigarette on RP[3] as previously reported, the preferred ring-tipped cigarette will be tested on a POL National mailout. The control will be a Marlboro 100 with the same tipping design as present on the ring-tipped experimental.

B. Reduced Delivery Virginia Slim Cigarettes

An experimental 100 mm Virginia Slim with an all-CA filter and Hauni dilution and 4 mg lower in tar than the current Virginia Slim was assembled. Evaluation by the Flavor Group indicated the reduced delivery Virginia Slim to be an acceptable candidate for further subjective testing versus the current Slim.[2]

An SEF hand-out test found the experimental Slim to be equally preferred to the control. RP[3] testing of this latest experimental Slim is planned for January, 1974.[3]

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Source: https://www.industrydocuments.ucsf.edu/docs/slxm0000

C. Cigarettes with Flavor Particles on Tow

Attempts to develop a cigarette with an active carbon-menthol filter by depositing starch-based menthol particles and carbon on the inner section of a dual filter have been unsuccessful. Although an acceptable amount of menthol is available in the filter, an insignificant amount (<0.1 mg) is delivered in the mainstream smoke. The cigarette is subjectively unacceptable. Apparently the menthol is adsorbed by the carbon as it is released to mainstream smoke.

No further work is planned at this time to develop the active carbon-menthol filter with the starch-based menthol powder.

RP[3] testing of 10-12 mg tar cigarettes with cherry and cinnamon particles on the inner section of a dual CA filter has been initiated.

D. Modified Parliament 85/80

HTI results indicated the modified Parliament 80 cigarette to be equally acceptable to Parliament 80 and non-Parliament smokers.

Final cigarette specifications are being written for the modified Parliament 80 and will be transferred to Manufacturing.

A test market is planned for the modified Parliament 85/80 cigarettes.

E. 10 mg Parliament Cigarette

A program has been initiated to develop a 10 mg tar Parliament cigarette similar in appearance and taste to the modified Parliament 85/80 cigarette. The filter systems being considered to achieve the 10 mg candidate are as follows:

1. High RTD CA with perforated hard mouth-piece paper and perforated tipping.
2. High RTD CA with perforated cigarette paper.
3. High RTD CA with Hauni pin dilution into the filter.
4. High RTD CA with Hauni pin dilution into the tipping over the tobacco rod.
5. CA/MT fluted plastic dual with dilution through perforated tipping into the CA.

All of the above models have been demonstrated on Parliament CFF rods. Plans call for a cost study on all five filter candidates to be completed by the end of December, 1973. Subjective testing through RP[3] is to be completed by February, 1974, with HTI testing on the preferred model(s) initiated in February.

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F. 85 mm Slim Marlboro Cigarette

The redesigned 85 mm Slim Marlboro was not equivalent in tar and puff count to the standard Marlboro 85. A remake of the 85 mm Slim Marlboro is underway to achieve equal tar and puff count.[6]

RP[3] monadic testing of both the standard 85 mm and Slim Marlboro will be completed in January, 1974.

II. Cigarette Component Investigations

A. Spheropore A and B

Further investigation into molding grooved filter rods of Spheropore A will be discontinued. Efforts to control the non-uniform shrinkage of the rods in the mold have been unsuccessful.

Evaluation of additional samples of Spheropore B from Monsanto indicate TPM efficiences ranging from 75-80% for both grooved rods and granular materials. However, the friability of these materials will have to be further improved.

A meeting with Monsanto personnel is planned for early January, 1974, to review the Spheropore materials.

B. B.F. Goodrich MP

Additional SEF testing of various batches of B. F. Goodrich MP material indicated no significant taste differences from batch to batch.[7] Currently, RP[3] testing of B. F. Goodrich MP material (70% TPM efficiency) is planned for January, 1974. For this test, all MP filters will be produced using the Baumgartner P-S-P filling unit (90% space-fill). A stiff plug wrap has been obtained for the Baumgartner unit to give acceptable rigidity to the MP filters.

Recently, B. F. Goodrich indicated some problems in batch to batch MP reproducibility due to changes in the starting raw materials. These problems are being investigated and should be resolved.

References

1. RP[3] Test Coded R-EPF-1
2. N.B. 6325 - 91

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Source: https://www.industrydocuments.ucsf.edu/docs/slxm0000

3. SEF Booth Test, "Va. Slim 100, X8D3BCD vs. 4 mg Reduced Va. Slim X8D3BCE", December 6, 1973.
4. N.B. 6316 - 31.
5. HTI Test of Experimental Parliament 80 vs. Production Parliament 80, Project #3044/3045.
6. N.B. 6325 - 94.
7. Test Results, MP Goodrich Coded G3MX and G3MW "C" vs. MP Goodrich Coded G3MV "E", December 5, 1973.

/mc

P. N. Gauvin

2022160829

Source: https://www.industrydocuments.ucsf.edu/docs/slxm0000

CHARGE NUMBER: 2105

PROJECT TITLE: Filter and Cigarette Process Development

PERIOD COVERED: November, 1973

PROJECT LEADER: J. M. Washington

DATE OF REPORT: December 12, 1973

I. Extrusion Systems Development

The extruder drive system failed electronically, and the cost of calling in a serviceman approached the cost of a new unit. The new unit was shipped and installed with minimum delay. Advantages of the new drive include built-in diagnostic circuits, ease of access to all components, and the reserve capacity to power a larger, higher speed extruder drive motor.

Modification of the Molins bed, including replacing the cutter, conveyor, internal and external gears, and the "ribbon" wheel, is now complete. Both regular (RF) and square-fluted (SF-18) filters were produced to all specifications, and length uniformity was demonstrated. The collapsed-tube puller for string-up has been proved satisfactory, and the plug dryer and garniture cooling systems are adequate.

Length gears are on hand to cut plug lengths of 90, 100, 108, 120, 114, and 126 mm.

II. FFM-5 Product Development

Using a modified glue pot system (heated), "star wheel" glue transfer, and the slotted contact roll gave sufficient operability to the Hot Melt Hauni operation to produce test samples. Cigarette samples had 60% dilution and 70% flute blockage. With dilution adjusted to 20% by blocking dilution holes, analytical results were:

FTC Tar	13.0
TPM	15.2
Nicotine	0.92
H_2O	1.3
P.C.	8.2

These values are nearly equivalent to Marlboro Lights.

Hot melts evaluated were Eastobond A-167S[1], Compounders, Inc. #4E449[2] and #4E469[3]. Further tests, both for processing conditions and hot melt trials will continue.

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III. Granular Space Fill Development

Process development has continued on the Baumgartner equipment. A very heavy combining wrap (0.005-0.006 inches) is required to prevent the collapse of granular filled space during smoking. With medium weight combining wrap available (0.004 inches) 85-90 percent space fill was achieved at 780 4-up plugs per minute.

A modified Vibra-Screw screw feeder has been designed to meter granular materials into the filling hopper, and a quote for $3704 with a ten week delivery has been received.

Further testing using a heavy combining wrap from Ecusta will be conducted during December.

References

1. Research Notebook No. 6360, p. 16, 17, 18
2. Research Notebook No. 6360, p. 19
3. Research Notebook No. 6360, p. 20

J. M. Washington

2022160831

Source: https://www.industrydocuments.ucsf.edu/docs/slxm0000

CHARGE NUMBER : Various

PROJECT TITLE : Flavor Development

PROJECT LEADER : Howard L. Spielberg

PERIOD COVERED : October 31 - November 30, 1973

DATE OF REPORT : December 11, 1973

I. Flavor Development

An RP^3 test has been requested to evaluate RL at 17½% with flavor fortification against a control model with BL. Additional RL models have been prepared using various levels of Dap and ammonia to alter subjective and analytical response. The two models listed below were made to October, 1973, Marlboro specifications, substituting RL for BL. Sample B contained Dap and ammonia added to the RL and blend respectively and Sample A has no additional Dap or ammonia.

	A	B
FTC	16.0	17.2
Nicotine	1.08	1.28
Puff Count	8.2	9.0

Differences in subjective response were also noted. Work in this area is continuing.

Evaluations of ES samples, cased with different humectants are underway. The stems were cased and cut at Westab and expanded at R&D. Preliminary analytical and subjective evaluations show no obvious differences between the ES samples. An RP^3 test is being prepared for shipment, evaluating a special ES casing against a control casing. The ES level in these models is at 7%.

An RP^3 test of specially cased EBS is being readied. This test model uses EBS at the 5% level replacing strip. Casings for EBS have been prepared for process development of EBS. Experimental models using EBS at 5% (excess) in total utilization blends have been prepared and are being evaluated. Flavored Burley RKS is also being reevaluated. Water extracted, O_3 treated EBS, evaluated at 8% level against washed EBS showed no obvious differences. Additional evaluations are underway. An RP^3 evaluation of washed vs. unwashed EBS has been requested. Models using 25% washed EBS with 75% Bright Strip and 25%

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unwashed with 75% Bright Strip have been prepared for evaluations. The washed model had an 85% reduction in nitrates and showed a 50% reduction in NO_4 delivery. No other obvious gas phase differences were noted. Subjective evaluation of these models is underway. Chemical treatments of EBS, including H_2O_2 followed by NH_4OH_1 are being conducted. Gas phase data on cigarettes using 10% H_2O_2 treated EBS show reductions acetaldehyde, isoprene and HCN.

A Marlboro Lights-type menthol cigarette with Hauni ventilation has been prepared for National POL testing against Salem. Alpine Slims vs. Alpine Regular 100's are being evaluated by RP^3. Several models of modified menthol cigarettes are currently being evaluated.

A 14 mg cigarette model was evaluated against a regular delivery model. The results were satisfactory. Two 12 mg models are scheduled for RP^3 testing against Marlboro type controls.

A cigarette model with 22% RKS and no ES, ET, RL or BL was prepared and is currently being screened.

Several humectants, including propylene glycol, glycerine, triethylene glycol, 1-3 butanediol and sorbitol, are being evaluated at various levels in cigarettes. Subjective and analytical evaluations are being conducted.

Evaluations of a flavored Rappaport Stem cigarette wrapper have been conducted. Boothe testing showed the control to be harsher and the experimental to be sweeter. Subjective screening indicated that the experimental was over flavored. Additional wrapper, with reduced flavor, is expected.

II. Flavor Development Assistance

Evaluations of DIET models are being conducted. Preliminary screening of DIET sample, at the 5%, 10% and 15% levels compared with Westab ET at comparable level, showed no obvious differences between samples. SEF is scheduling a series of evaluations of DIET models.

Odor screening of pipe tobacco, packaged in modified cans, was conducted. The breathable cans showed less fermented, winey character than the regular sealed cans.

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New flavors have been developed for evaluation on LTF models. New models are being evaluated.

Flavors have been supplied for all bright cigarette models. These models are being evaluated. Flavors were sent to affiliates in Dominican Republic and New Zealand. Subjective screening of 17 Russian cigarette brands was also conducted.

III. References

6326	53-90
6424	13-56
6305	94-100
6395	20-51
6293	81
6376	53-62
5145	35
5367	27
6403	52-73

H. L. Spielberg

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CHARGE NUMBER : 2306

PROGRAM TITLE : Flavor Transfer

PERIOD COVERED : October 26 - November 25, 1973

WRITTEN BY : R. M. Ikeda

DATE OF REPORT : December 4, 1973

I. Menthol Transfer[1]

The three studies for the optimum level of triacetin (TA) for percent menthol delivery were completed. Graphically the data shows that 3.5-6.3% TA on filters yields the highest percent menthol delivery. Triethylene glycol diacetate (TEGDA) was used in place of TA and results showed decreasing menthol delivery with increasing TEDGA content. Statiscal analyses were done by Mr. J. Tindall and the results confirm the graphical conclusions.

II. Subjective Quality of Smoke[2]

A modified GC injection port which will permit sweeping an entire TPM pad was completed and installed. This sampling procedure for TPM will allow us to study the subjective quality of TPM constituents.

III. Factory Flavors[3]

The work on factory flavors was completed and a final report is being written.

IV. References

1. 6345 51-56
2. 6271 99
3. 6193 52-55
   6271 84, 87-89

R. M. Ikeda

R. M. Ikeda

/ec

2022160835

Source: https://www.industrydocuments.ucsf.edu/docs/slxm0000

CHARGE NUMBER: 2500

PROGRAM TITLE: SYNTHESIS OF TOBACCO ADDITIVES

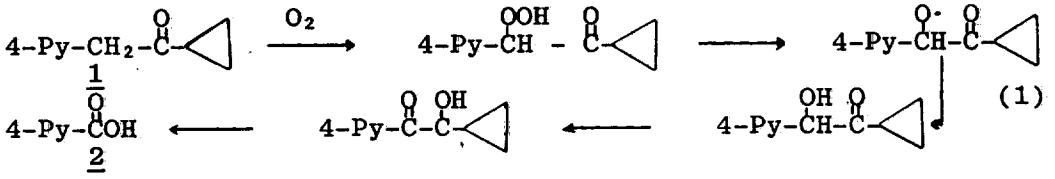
PERIOD COVERED: OCTOBER 31 - NOVEMBER 30, 1973

PROJECT LEADER: T. B. SANDERS

DATE OF REPORT: DECEMBER 4, 1973

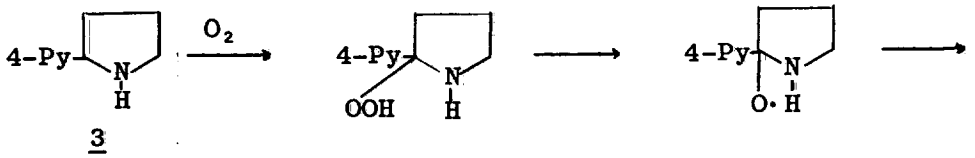
I. NICOTINOID CHEMISTRY

Several interesting reactions have been observed regarding both 2-and 4-picolyl cyclopropyl ketones. For instance, the only product isolated, albeit in low yield, from the attempted preparation of the N-oxide of 2-picolyl cyclopropyl ketone was picolinic acid. Under the same conditions, however, *i.e.*, m-chloroperbenzoic acid in chloroform at 0°, both 3-and 4-picolyl cyclopropyl ketones give almost quantitative yields of the corresponding N-oxides.[1] The most likely explanation would seem to be a rearrangement *via* a cyclic transition state formed by interaction of the oxygen of the initially formed N-oxide with the carbonyl group of the ketone. Similar reactions have been observed in analogous systems.[2] More difficult to understand is the recently observed partial conversion of 4-picolyl cyclopropyl ketone (1) on standing to isopicolinic acid (2)[1]. In all probability this reaction proceeds by way of a free radical oxidation (Equation 1) and is related to the spontaneous conversion of 4-nornicotine (3) to 4-myosmine (4) (Equation 2).

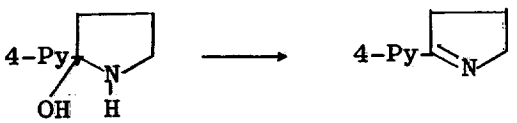


(1)

1 2



3



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Source: https://www.industrydocuments.ucsf.edu/docs/slxm0000

Apparently a carbon flanked by both a 4-pyridyl group and another activating group is subject to facile hydrogen abstraction. These reactions are under investigation.

The synthesis of 1-tosyl-3-(3-pyridyl)-azetidine (CR-1321) has been accomplished in five steps starting from ethyl 2-(3-pyridyl)-acetate. Ethyl 3-methoxyimino-2-(3-pyridyl)-propionate (CR-1320), the second intermediate has been added to the CR-file. The key step in this reaction sequence is the use of CR-1320 as the precursor of the corresponding amino alcohol. The use of the methoxyimino compound rather than the hydroxyimino compound (oxime) prevents cyclization to the isoxazalone which cannot be reduced to the desired amino alcohol. This scheme is now being extended to the synthesis of 2-(3-pyridyl)-azetidine since, if successful, it represents a significant improvement over the original scheme. At the present time, ethyl 2-nicotinylacetate has been prepared from the base catalyzed condensation of ethyl acetate with ethyl nicotinate and has, in turn, been converted to the O-methyl oxime. The reduction of the O-methyl oxime is under study at this time.[3]

Work is continuing on tying up the loose ends of the 5'-cyanonicotine problem. The Murphy reaction[4] was repeated four times in order to obtain a sufficiently large sample of 2'-cyanonicotine for positive identification. The dehydration of *cis*-5'-carboxamidonicotine was tried with phosphonitrilic chloride, a new reaction.[5] No nitrile was obtained.[6] Hydrolysis of the mixture of *cis*- and *trans*-5'-cyanonicotine in methanolic HCl gave a mixture of the corresponding 5'-carbomethoxynicotines. The mixture of esters could be partially resolved using column chromatography on silica gel. Column chromatography will also be explored on the mixture of the nitriles.[7]

Work on the nicotine substituted on other positions of the pyrrolidine ring has begun. The synthesis of *trans*-3-carboxycotinine[8], was successfully carried out *via* the condensation of methyliminonicotinaldehyde with succinic anhydride.[6] Attempts to build up a substituted nicotine *via* a Hoffman-Loeffler type cyclization have so far proved unsuccessful in that reaction of methyliminonicotinaldehyde with propyl magnesium bromide, the first step, was unsuccessful.[7]

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II. FLAVOR-RELEASE COMPOUNDS

The pyrolysis of ethyl 2-methyl-3-phenyl-3-hydroxy-propionate was investigated over a temperature range of 177-300° using gas chromatography-mass spectrometry. It has been shown that the ester decomposes both *via* dehydration to give ethyl 2-methyl-*cis*-and *trans*-cinnamate and a six-center fragmentation to give benzaldehyde and ethyl propionate. Quantitative data were not obtained. The custom synthesis of 300 g of poly(1-menthyl 1,1-dimethyl-allyl carbonate) was initiated. At this time 100 g. has been prepared and delivered to Development.[9]

III. REFERENCES

1. W. B. Edwards 6320
2. E. Hayashi and E. Oishi, J. Pharm. Soc. Japan, 87, 940 (1967).
3. H. Secor 6377
4. P. J. Murphy, J. Biol. Chem., 248, 1796 (1973).
5. J. C. Graham and D. H. Murr, Can. J. Chem., 50, 3857 (1972).
6. J. F. DeBardeleben 6437
7. E. B. Sanders 6412
8. M. Cushman and N. Castignoli, Jr., J. Org. Chem., 37, 1268 (1972).
9. E. G. S. Rundberg 6442

/slb

Edward B. Sanders

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Source: https://www.industrydocuments.ucsf.edu/docs/slxm0000

CHARGE NUMBER: 2501

PROGRAM TITLE: NUCLEAR AND RADIOCHEMISTRY OF SMOKE

PERIOD COVERED: November 1-30, 1973

PROJECT LEADER: R. W. Jenkins, Jr.

DATE OF REPORT: December 10, 1973

AN ANNUAL REPORT WAS WRITTEN THIS MONTH.

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CHARGE NUMBER: 4008

PROGRAM TITLE: Smoker Simulation Studies

DATE OF REPORT: December 10, 1973

PERIOD COVERED: November 1 - November 30, 1974

PROJECT LEADER: George C. Kiritsis

I. Data Collection

A. Dictaphone Recorder

Three old recorders were returned to Dictaphone Corporation for head changes. These recorders have been returned and are being evaluated for performance.

B. Sony Recorder

The Sony recorder, used as a playback unit, has performed well in our laboratory. Two additional units have been received and are being evaluated. One of these units will be placed in the computer room for evaluation as a playback unit into the computer.

C. Smoker Profile "Recording Units"

The six new units received from the Engineering Group are being evaluated and calibrated prior to placing in the field for use in product testing.

D. Methods of Filtration Study

Nineteen subjects have completed smoking all four cigarette models and their individual data has been read-out by the computer.

Individual subject data collection for this study has been completed. Puff by puff TPM on each of the four models was obtained using a Command Tape from an individual smoker. In addition, a statistical summary of the various smoking parameters of all nineteen subjects on each of the four models was compiled and analyzed. Preliminary evaluation of the data indicates (1) that the cigarettes were not smoked alike by the panelists; (2) the smoking pattern appeared to be most greatly affected by dilution; (3) TPM (and nicotine) level to the smoker is not directly related to subjective response.

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Source: https://www.industrydocuments.ucsf.edu/docs/slxm0000

II. Human Smoking Simulator

During this reporting period, the variables which could affect the calibration results on the Simulator were examined; that is, the length of the vacuum tubing between the various apparatus; the amount of vacuum; and the RTD of the dummy cigarette and the test port.

A standard procedure has been established for calibration purposes.

III. Plans

A. Continue the monthly calibrations on the smoking profile recording units, human smoking simulator and Hastings flow meter.

B. Complete evaluation of the Sony recorder as a play back instrument.

C. Prepare a command tape to reproduce the average smoking pattern of the total number of panelists on each model in the "Methods of Filtration" study.

D. Pursue studies to correlate cigarette characteristics with subjective response and smoking patterns.

IV. References

Notebook 6405, pp. 31-36

George C. Kiritsis

George C. Kiritsis

2022160841

Source: https://www.industrydocuments.ucsf.edu/docs/slxm0000

CHARGE NUMBER: 4009
PROGRAM TITLE: Smoke Modification
PERIOD COVERED: November, 1973
PROJECT LEADER: W. A. Geiszler
DATE OF REPORT: December 11, 1973

1. "Cherry Sidestream" Cigarette

RP[3] testing of a cigarette containing a "subtle" level of CR-1214 (0.1 mg/cigt.) indicates that panelists can detect taste and aroma differences in the treated cigarette versus an untreated control, but they prefer the control.[1] Another subjective test is planned at a higher CR-1214 level where sidestream aroma modification is more pronounced.

2. Salt Additives on Cigarette Paper

Sodium sulfate added to cigarette paper has shown promise of giving good cigarette ash appearance without reducing puff count as much as sodium citrate. Further testing is needed to confirm this behavior at sodium levels corresponding to 0.6-1.0% citrate, the range presently used on commercial products.[2]

A study is underway to understand phosphate-treated cigarette papers. The study will include sodium, potassium and ammonium phosphates in mono-, di-, and tri-basic forms. A comparison of mono- and di-basic potassium phosphates confirms that the di-basic salt is a better burn rate promoter, but both salts give faster cigarette burn rates than untreated paper.[3]

3. Cigarette with Guar Wrapper

A guar-coated cigarette paper containing about 5% guar and having a Greiner porosity rating of 73 seconds has given a 10% increase in puff count and 20% increase in FTC Tar over an untreated control.[4] The coating will be tried on papers containing no burn additive, looking for good ashing properties and a reduced cigarette burn rate.

References

1. Memo, D. F. Reynolds to W. A. Geiszler, November 26, 1973
2. NB 6455, p. 1
3. NB 6439, p. 2
4. NB 6455, p. 3

W. A. Geiszler

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CHARGE NUMBER: 6906

PROGRAM TITLE: BIOLOGICAL EFFECTS OF SMOKE

PERIOD COVERED: NOVEMBER 1-30, 1973

PROJECT LEADER: R. A. PAGES

DATE OF REPORT: DECEMBER 6, 1973

I. TISSUE CULTURE

A new experimental series was begun in which cells of the F1706 line derived from Fischer rat embryos[1] were treated with 50 µg/ml each of 3-methylcholanthrene, 1R1 WSC, and X6DOAD WSC. The treated cells are being passaged every week and are being monitored for the appearance of macroscopic foci of cells, indicative of transformation.[2]

Work was begun to familiarize ourselves with the techniques necessary to preserve cells by freezing and storage in liquid nitrogen. The purpose of this work is to provide us with a continuing source of cells inhouse and thus to free us from total dependence on outside sources of supply.[2]

II. MUTAGENESIS ASSAY

Work was continued on the establishment of stock cultures of several tester strains of S. typhimurium. Preliminary tests are underway with several mutagens whose modes of action are known (ethyl methanesulfonate, 2-aminopurine, and hycanthone monomethanesulfonate) in order to establish conditions for quantitatively monitoring the responses of the tester strains.[4]

III. PARTICLE NUMBER AND SIZE DISTRIBUTION OF TOBACCO DUST

Tobacco dust samples submitted by Norman Rowe were analyzed on the Coulter counter. It was determined that the instrument could size and count tobacco dust particles in the ranges encountered in the BL and RL facilities.[5]

IV. E. COLI TOXICITY EVALUATION BY CELL NUMBER ANALYSIS

A. EFFECT OF TRITON X-100

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Triton X-100, the detergent used to enhance solubilization of whole smoke condensates in the E. coli differential toxicity assay, was shown to affect the polA+ strain only. A shift of the mean size of the population to a smaller size was observed. This observation may be related to the higher variability of the polA+ strain in the assay (see below).[5]

B. EFFECT OF METHYL METHANESULFONATE (MMS)

The time course of the effects of MMS treatment on both the polA+ and polA- strains was monitored on the Coulter counter. It was concluded that some of the effects of MMS can be monitored by both cell number and size distribution.[5]

C. PHAGE LYSIS OF CELLS TREATED WITH MMS

Investigation of the possibility of distinguishing "live vs. dead" cells following infection with T2 phage was continued. The data show trends in the expected direction, *i.e.*, 50 percent of the polA- strain was affected by MMS treatment. However, more work needs to be done to ascertain the validity of this approach.[5,6]

V. BIOASSAY (DIFFERENTIAL TOXICITY IN E. COLI)

An experimental series involving seven operators was run on five consecutive days to try to establish the precision of the assay. Statistical evaluation of the data supports the following conclusions:

A. There is no significant difference between the precision of the assay as it is now run and the assay as originally described.[7] (differences exist only in the dilution and plating technique).

B. There is more variation in the polA+ strain than in the polA- strain (±23% vs. ±13% for one operator on one day).

C. For three operators on one day the difference between the toxicities of the positive strain (%P)

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and the negative strain (%N) must exceed 16 to be significant.[8]

A preliminary experiment was performed to compare viable cell counts on agar plates with particle counts obtained on the Coulter counter. The data suggest that for both strains 15-20% of the cells are nonviable at the time of treatment. This could be due to the fact that the cells are in an early stationary period of growth when some cells must be dying.[6]

We are currently characterizing the strains used in the assay to assure ourselves of their integrity.[6,8]

VI. REFERENCES

1. Freeman, A. E., *et. al.*, (1973), J. Nat. Cancer Inst., 51, 799-808.
2. Thompson, L. 6433/25-26
3. Weissbecker, L. 6454/8
4. Glenn, A. 6453/3-4
5. Neal, W. 6363/68-69
6. McCuen, R. 6441/13-14
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2022160845

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CHARGE NUMBER: 6908

PROGRAM TITLE: SMOKE CONDENSATE STUDIES

PERIOD COVERED: OCTOBER 30 - NOVEMBER 28, 1973

PROJECT LEADER: W. F. KUHN

DATE OF REPORT: NOVEMBER 30, 1973

I. CONDENSATE PREPARATION

Whole smoke condensate (WSC) from selected cigarette types was processed on schedule for Project 6900. The WSC from all-Bright tobacco cigarettes containing the alkaloid-free hexane extractables from radioactive (^{14}C) Bright tobacco was processed.

II. CHEMICAL AND BIOLOGICAL ANALYSES

A. CHEMICAL ANALYSES

The study of chemical stability of WSC as a function of storage temperature and physical state was completed. In this experiment, it was concluded that the amounts of nicotine, phenols, total aldehydes and total nitriles do not change over a 53-day time interval. These chemical parameters are not affected by storage of WSC in acetone at 6°C or at -20°C.[2]

The gas chromatographic profiles of the petroleum ether extractables from TPM for cigarettes made by different reconstitution processes (TFP, Schweitzer and Rapaport) were obtained. With one exception, these profiles were similar, although minor to moderate differences in the ratios of several peaks were observed. The profile obtained from the TFP sample showed three major peaks which did not appear in the other profiles. The origin of these components is under investigation.[4]

The attempts to separate hydroxyacetone from dimethylnitrosamine on four different gas chromatographic columns were not successful. A smoke sample has been sent to the Department of Agriculture for high resolution mass spectrometric analysis for confirmation of the dimethylnitrosamine.[1]

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The first attempt to quantify the dimethylnitrosamine (DMNA) resulted in the following observation: about four times as much DMNA is present in the dry ice/acetone trap as is found in the sodium hydroxide trap used to collect "fresh" smoke. This observation will be confirmed.[1]

A high speed liquid chromatographic separation scheme was developed using a Zorbax Sil column. This scheme separates the polynuclear aromatic hydrocarbons (PAH's) from other UV-absorbing compounds in fraction VI (Grimmer scheme).[5]

The separation efficiency of Sephadex LH-20 in both an analytical and preparative mode has been established for equivalent fractions of WSC from an expanded Bright tobacco cigarette and its control. This technique is successful in separating the terpenoids from the PAH's and degradation products of chlorinated pesticides.[1,3]

Work continues on the nitrogen profiles from selected cigarette types. These profiles are similar with the exception of that obtained from an LTF cigarette type. Current effort is directed toward the identification of these nitrogen-containing compounds.[6]

It was demonstrated that rechromatography of fraction VI (Grimmer scheme) on deactivated silica gel followed by preparative thin layer chromatography resulted in a gas chromatographic profile which yields nearly baseline separation of the majority of components in this subfraction.[1]

The Grimmer separation scheme was completed on WSC from all-Bright tobacco cigarettes containing not only the total hexane extractables from radioactive (^{14}C) Bright tobacco but also the radioactive hexane extractables minus the tobacco alkaloids. The mass distribution data from these two experiments were reasonably reproducible. The accountability of the radioactivity seemed reasonable; *i.e.*, the activity was reduced in the second experiment for those fractions known to contain the tobacco alkaloids. A special report covering the details of these experiments is being drafted.[2]

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B. BIOLOGICAL ANALYSES

The study of stability of WSC in relation to its biological activity (E. coli procedure) through 53 days of storage was completed. According to the results of this bioassay it was concluded that there is no change in the activity of the WSC through the 25th day but the activity on all samples drops on the 39th day. The results on the 53rd day (termination date of the test) are unexplainable because all the test samples including the positive control were inactive.[2]

III. REFERENCES

1. N. Einolf 6368, 64
2. F. Gager 6448, 31
3. T. Katz 6127, 96
4. R. Levins 6440, 9
5. G. Smith 6414, 29
6. E. Trusdell 6231, 96

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Source: https://www.industrydocuments.ucsf.edu/docs/slxm0000

CHARGE NUMBER : 8205

PROGRAM TITLE : Packaging Development

PERIOD COVERED : October 16 - November 15, 1973

WRITTEN BY : W. I. Carr

DATE OF REPORT : December 3, 1973

I. Alpine 100's Flip Top Boxes

Wider package end sealer bars, recently installed on the overwrap machine, were used for the latest test market production run. Also, a machine trial run was made with polyethylene coated blanks. The adhesive used (Fuller #3544) appeared to be adequate for all joints except the 180° lid flap. Additional trial runs will be made when the adhesives ordered for the 180° lid flap are received.

Storage tests of both packages are being conducted.

II. Storage of Cigarettes in Freezer Spaces (0°F. to -10°F.)

We have been informed that there is more freezer space (0°F. to -10°F.) than cooler space (34°F. to 45°F.) for long term storage of cigarettes. Since the Company, except for one week or so, has had no experience with freezer storage of cases of cigarettes; arrangements have been made to store cases of cigarettes in a freezer space in the Richmond Cold Storage Warehouse for a period of six months. Cases will be removed on a monthly basis for examination.

III. Substitutes for Foil/Paper Liners in Soft Packs

A preliminary evaluation of substitute materials for the aluminum foil paper laminate liner in soft packs indicate that one candidate from Nicolet (#60) and the one from Milprint made up as finished packages performed in hot-dry storage tests at least as well as aluminum foil. Further improvement in machinability of the materials is expected.

IV. References

Book	Pages
6388	95-98

W. I. Carr

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CHARGE NUMBER: Various

PROGRAM TITLE: New Process Investigation

WRITTEN BY: G. L. Mathe

DATE OF REPORT: December 15, 1973

I. Ultrasonics

Based upon previously reported experimental results, a laboratory type, variable frequency ultrasonic generator system was selected and ordered from Fibra-Sonics, Inc., Chicago, Illinois. Delivery is expected late this month.

Special ultrasonic transducer specifications are established and one unit ordered for the development of the high density SCS type filter processing program. Several basic type transducers will be available from Fibra-Sonics, Inc., for feasibility studies, and investigations on several concepts in ultrasonics, i.e., plastic filter tube sealings; shattering of compacted MP material; carbon fallout, etc.

II. Microwave-Fused (Wrapless) Plug Development

Modification and adoptation of a variable speed tow puller is completed and tried. Microwave guard, featuring exchangeable ceramic tubings of various diameters, is mounted on the puller unit along with a special rod cooler.

Connection of the puller - microwave guard to the microwave generator system is underway. It is expected that the first trial of rapid fusing of the tow fibers may be conducted during the week of December 17.

III. Plug Space Plug Extrusion

A special extruder die, designed for the feasibility study of "plug combining by extrusion" program, was fabricated by Gatto Machinery Development Corp., Hanpauge, New York, and delivered to R&D. Adaptation, tryout and debugging of this equipment is tentatively scheduled for the first half of January. In the meanwhile, a bread-board feeding system is being attached to the extrusion line to evaluate concept and establish production specifications.

G. L. Mathe

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